

PATENT
213/001-CIP

residue purified by chromatography (CH₃OH- Hexanes-CHCl₃, 1:1:8) to yield 414 mg (45%) of 5'-O-(4,4'-dimethoxytrityl)-3'-C-aminomethylthymidine as a colorless solid.

5

Example 10
Preparation of

5'-O-(4,4'-dimethoxytrityl)-3'-C-trifluoroacetamidomethylthymidine

10 A solution of 5'-O-(4,4'-dimethoxytrityl)-3'-C-aminomethylthymidine (361 mg; 0.628 mmol) and ethyl thiotrifluoroacetate (490 mg, 3.12 mmol) in anhydrous THF (6 ml) was stirred at room temperature for 6 h. Solvent was evaporated and the residue purified by chromatography on 15 silica (5% CH₃OH in CH₂Cl₂) to yield 411 mg (98%) of 5'-O-(4,4'-dimethoxytrityl)-3'-C-trifluoroacetamidomethylthymidine as a colorless powder.

20

Example 11
Preparation of

5'-O-(4,4'-dimethoxytrityl)-3'-C-trifluoroacetamidomethyl-thymidine 3'-(2-cyanoethyl-N,N-diisopropylphosphoramidite)

25 To a stirred solution of 5'-O-(4,4'-dimethoxytrityl)-3'-C-methylthymidine (411 mg, 0.614 mmol) and diisopropyl-ethylamine (0.64 ml, 3.65 mmol) in anhydrous dichloromethane (6 ml) at 0 °C under argon was added dropwise a solution of 2'-cyanoethyl-N, N-diisopropylchlorophosphoramidite (410 mg, 1.83 mmol) in anhydrous dichloromethane. The resulting reaction mixture was stirred at room temperature for 2 h, cooled to 0 °C, diluted with cold CH₂Cl₂ (30 ml), and washed with cold NaHCO₃ (3 x 20 ml). The organic layer was dried over Na₂SO₄ and concentrated. The residue was purified by